ON THE STAINING OF FELDSPATHOIDS, AND ON ZONAL STRUCTURE IN NEPHELINE

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The use of acids for distinguishing certain rock-forming minerals is older than the use of the microscope for this purpose. It has long been known that nepheline and a few other silicates are readily decomposed by acids, and that gelatinous silica is formed by the reaction. H. Behrens 1 seems to have been the first to suggest the idea of making the silica gel visible by impregnating it with dyestuff, but he gave no detailed account of his procedure. E. Boricky, 2 who devised many ingenious methods of microchemical analysis, used chlorine gas to decompose the silicates and stained the resulting silica gel with fuchsine solution. All the soda-feldspathoids and even olivine could be stained in this way, but not leucite. F. Zirkel 3 describes briefly how the thin section is to be covered with a thin film of hydrochloric acid and then, after washing, immersed in a weak aqueous solution of fuchsine or methyl violet. Similar directions are given in other text-books, but often the instructions are so vague that one wonders whether the author ever practised the method himself or just related it at second hand. For instance, E. Weinschenk 4 says the section that has been gelatinized by acid should be immersed "some hours" in congo-red or malachite green; yet anybody who has ever stained feldspathoids must know that it is not necessary to leave the preparation in the dye for more than a minute. Nobody seems to have given much attention to the effects produced by acids other than hydrochloric, or to the method of preserving the stained section as a permanent preparation. In order to make the fullest use of the staining method, these points deserve attention.

It is easy to satisfy oneself that liquid hydrochloric acid is not the most suitable acid for the purpose, because much of the silica goes into solution and is washed away. This difficulty can be overcome by exposing the rock section to the fumes of concentrated HCl instead of the liquid; excellent preparations can be made in this way, but it is difficult to restrict the action to a particular part of the section. Strong sulphuric acid and strong nitric acid are excluded because of their action on Canada balsam, and weak solutions of these acids are open to the same objection as hydrochloric acid. But a saturated solution of potassium bisulphate is a satisfactory reagent; it attacks nepheline and gives a thick gel in a few minutes. It has no action on leucite. A saturated solution of oxalic acid also dissolves nepheline but gives only a thin deposit of silica gel.
This acid acts slowly on leucite and forms enough gel, in 30 to 60 minutes, to take the stain quite deeply. It also acts on lime-rich plagioclase, though more slowly than on leucite.

The most useful reagent of all, for the soda-feldspathoids, is syrupy phosphoric acid (U. S. Pharmacopoeia, 85%). A thin film of the acid is spread with a glass rod over the part of the section which it is desired to stain. After about three minutes the slide is gently dipped in water to remove the acid and is then immersed in a weak solution (0.25% is sufficient) of methylene blue for about one minute. Nepheline, sodalite and analcime are stained deep blue by this treatment; melilite takes a lighter blue; leucite is not affected. After staining it is still possible to distinguish nepheline from sodalite or analcime by its birefringence. Other dyes may be used, for instance malachite green or methyl red; but the very soluble red dyes such as fuchsine and saffranine are not suitable for making permanent preparations as they diffuse through the mounting medium.

After the section has been stained, the next problem is to preserve it. The procedure recommended by Weinschenk and others is to let it dry and then mount in xylol-balsam. This is quite unsatisfactory because in drying the silica gel shrinks, cracks, and breaks up to such an extent that the shape of the gelatinized grains may be lost. A simple way of overcoming this is to remove the section from the dye, dip it a few times in water to remove the excess of dye, and immediately put on a drop of ordinary liquid glue and a cover-slip. (I have used Dennison’s glue and Le Page’s glue, but any thick solution of glue in water would be suitable.) The glue should not be too watery or the dye will diffuse outwards and make the edges of the grains feathery. The glue dries quickly and the silica gel is fixed without shrinkage or disturbance. Preparations made in this way are quite transparent and will last for months in a moderately humid atmosphere; if a longer life is desired the cover-slip should be sealed with asphalt paint.

An alternative method, by which it is possible to cover the section with Canada balsam, takes advantage of the observation that the dyes named above, although insoluble in xylol, are soluble in a mixture of xylol and alcohol. After the section has been gelatinized, it is dipped in alcohol, then immersed in a solution of methylene-blue in equal parts of alcohol and xylol; after a minute it is dipped in pure xylol to remove the alcohol as well as the excess of dye, and then it is covered with a drop of xylol-balsam and a cover-slip. Although this method gives permanent preparations, they are not so perfect as those mounted in glue because some shrinkage can not be avoided.

When using the staining method to search for feldspathoids in a rock-
section, it is necessary to remember that zeolites are gelatinized too and that colloidal weathering-products of any kind will take up the dye. Thus it is useless to stain weathered rocks, and unless the rock is perfectly fresh it is unsafe to draw conclusions about the nature of a scanty interstitial material which takes the stain.

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The presence of a zonal structure in nepheline crystals was recorded by E. Boricky in 1874, and a good example was figured from Waschholderberg near Teplitz. In 1926, F. Becke and J. E. Hibsch gave a careful description, with many drawings, of zonal nepheline crystals in the phonolites of Bilin and Brüx, mentioning especially the Brüxer Schlossberg and Rudelsdorf. Most of these crystals show a core of normal refraction and birefringence followed by a narrow zone of weaker refraction and birefringence, and outside that a narrow mantle with higher refraction but weak birefringence. There may be an alternation of many such zones. The explanation offered by Becke and Hibsch was based on N. L. Bowen's study of the system nepheline-anorthite. Bowen showed that the presence of anorthite in solid solution causes the birefringence of nepheline to decrease to zero, after which it increases again with opposite sign. Becke and Hibsch concluded accordingly that the zoning described by them is a consequence of rhythmic variation in the anorthite content of the nepheline crystals, the zones of lower birefringence being those with more anorthite. O. H. Ödman in 1930 described zonal nepheline in rocks of Mt. Elgon, East Africa, and adopted the same explanation.

On applying the staining method described above to the phonolite of the Brüxer Schlossberg, I found that phosphoric acid has a selective action on the zoned nepheline crystals, the zones of lowest birefringence being almost unaffected while the rest of the crystal is deeply stained (Fig. 1). In a further series of tests I found that nepheline crystals which appear to be quite homogeneous in polarized light will sometimes exhibit a complicated zonal structure when stained by the phosphoric acid method. This was observed especially in the phonolite of Leeuwkraal, South Africa, and in a phonolite from Kenya which Bowen and Ellestad have described.

Since the zoning of nepheline may have any one of three explanations, namely presence of lime, presence of excess of potash, or presence of excess of silica (in other words, solid solution of anorthite, of kaliophilite, or of albite), it occurred to me that I might be able, by staining, to distinguish between these three possibilities. Fortunately for my purpose,
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Fig. 1. Zonal nepheline crystals in the phonolite of the Brüxer Schlossberg, Bohemia, after staining with phosphoric acid and methylene blue. Magnification 50. Note tiny zoned nephelines in the groundmass.

Fig. 2. Zonal nepheline crystals in the phonolite from Nyeri, Kenya, after staining. Before staining these crystals appeared to be homogeneous. Magnification 8.
Bowen and Ellestad have recently described and analyzed a potash-rich nepheline, in leucite-nephelinite from Lake Kivu; and an albite-rich nepheline, in phonolite from Kenya. Dr. Bowen was so kind as to lend me his specimens of these rocks for examination, and also to supply a specimen of synthetic nepheline containing 30% of anorthite. I had thin sections cut from these, and then stained them with phosphoric acid and methylene blue as described above. The results were as follows:—

1. The anorthite-rich nepheline, having the composition Ne$_70$An$_30$, was stained completely and deeply.

2. The potash-rich nepheline, having the composition Ne$_57$K$_{33}$An$_4$Ab$_1$, was stained completely and deeply, without any appearance of zoning.

3. The albite-rich nepheline, with the composition Ne$_68$Ab$_21$K$_{12}$An$_4$ developed a complicated system of stained and unstained zones (Fig. 2). In general the outermost zone was uncolored, as in the Bohemian phonolites.

It is clear, therefore, that neither anorthite nor kaliophilite is the cause of zoning in nepheline crystals, and that albite is the villain of the piece. More correctly expressed, the zoning of nepheline crystals is due to the rhythmic deposition of layers of nepheline-substance alternately richer and poorer in silica. All the rocks in which zonal nepheline has been observed contain much alkali-feldspar and have a relatively high silica content. The Kenya phonolite has 54.8% of silica; the Leeuwerkraal phonolite 58.0%; and two analyses of Bohemian phonolites, quoted by Becke and Hibsch, show 55.8% and 56.5%, respectively. Only one of the latter is said to contain zonal nepheline, but it is rather likely that zoning would appear in the other too if it were stained. The nephelinite from Lake Kivu is free from feldspar and its silica content is only 37.7%; the nepheline in it is not zoned.

Unexpected confirmation of the conclusion stated above, regarding the cause of zoning, appeared when a section of the Schlossberg phonolite was exposed to the action of hydrochloric acid gas and then stained with methylene blue. The usual relation was now reversed, the outermost zones of the nepheline crystals taking the stain more deeply than the cores, presumably because a thicker film of silica was formed over the albite-rich zones.

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